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Buried & surface cofiring compatibility of DuPont 5092D PTC thermistor composition with DuPont 951 LTCC

Abstract. This reports documents qualification tests made with the 100Ω PTC resistive composition DuPont (DP) 5092D, co-fired on the surface and inside (buried) DP 951 LTCC material, using a wide range of Ag & alloyed Ag-Pd conductor termination: DP 6142D & 6145 (Ag), and 6135D & 6143 & 6146 & 7484 (Ag-Pd). These tests were deemed necessary, as previous ones [1], carried out with Ag terminations only, were deemed not conclusive and reliable enough. The resulting resistors were electrically characterised, and the reliability of the different samples was analysed. Besides, the degree of deformation and morphology of the samples was qualitatively evaluated.

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Keywords : test patterns; LTCC; PTC resistors

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1. Introduction

Qualification tests of both resistive compositions used for LTCC projects, DP CF011 (10 Ω) and DP 5092D (100 Ω PTC), with DP 951 LTCC and Ag terminations have been carried out before [1]. The results (Figure 1) were satisfactory for DP CF011, but reliability was poor with DP 5092D.

The problems with DP 5092D was ascribed to reactions occurring between the Ag terminations (especially DP 6142D; DP 6145 was somewhat better) and DP 5092D, leading to the formation of an insulating zone (open circuit). Note, however, that some reaction is apparently beneficial in buried PTC resistors, as Ag [1] and Ag-Pd [2] conductors counteract bubble formation, which occurs when the resistor materials are buried alone or with (unreactive) Au terminations.

As the results for DP 5092D with Ag were deemed not reliable enough, a more extensive study was undertaken, including Ag-Pd conductors. One such conductor was tried before [3], but tended to generate more deformation of the LTCC. Therefore, a more complete series is tested in the present study.

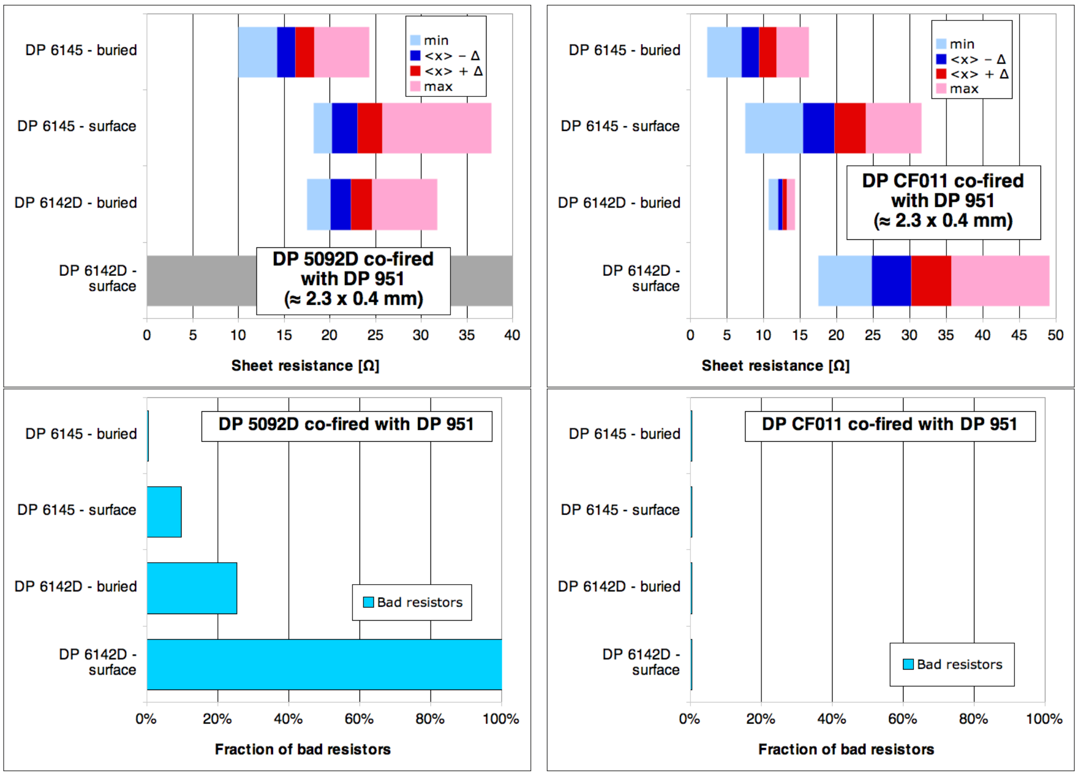

Figure 1. Summary of results for DP 5092D (left) and CF011 (right) from previous study [1]: sheet resistance (top) & reliability (bottom).

2. Experiments

The tests were carried out the same way as before [1]: a batch of resistors (ca. 2.3×0.4 mm^2) was screen printed onto an LTCC unfired substrate, and covered with an additional sheet cut out in such a way as to cover half of the resistors (Figure 2), in order to make a series of co-fired surface (left) and buried (right) resistors. The list of tested materials is given in Table 1. In most cases, two substrates (hereafter labelled A and B) were fabricated.
The samples were fired using a typical LTCC profile, with a debinding step around 450°C, followed by a nominal 5 K/min ramp to peak and 875°C 30 min dwell[1]. The resistor values were measured, and the samples checked for deformations and other signs of incompatibility between materials, with thermal characterisation being performed on some samples.

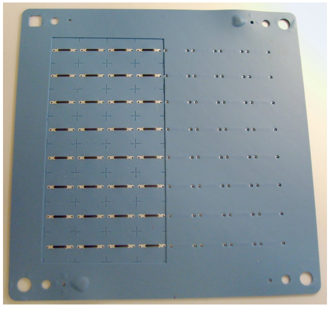

Figure 2. Test samples [1].

Composition	Type [4]
DP 951	LTCC tape
DP 5092D	100 Ω PTC thermistor, designed for alumina
DP 6142D	Ag, signal lines + capacitor electrodes
DP 6145	Ag, signal lines
DP 6135D	Ag-Pd, nominally post-fire
DP 6143	Ag-Pd, nominally post-fire, solderable, replacement for DP 7484
DP 6146	Ag-Pd, co-fire only, solderable
DP 7484	Older composition, designed for alumina but works with LTCC

Table 1. Tested materials.

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3. Results and discussions

3.1. Sheet resistance and reliability

The sheet resistance of the different samples is given in Figure 3, together with the observed reliability. The latter "fraction of bad resistors" is defined somewhat arbitrarily as those who considerably deviate from the mean value, but, in most of these cases, resistance is indeed very high, i.e. > 100× the normal value. Table 2 summarises the results of optical observations. The different samples may be separated into two groups:

1) Samples fired with DP 6135D, 6143 and 6146 (sample A) exhibit a "base" sheet resistance lying around 15 Ω, surprisingly similar for both surface and buried resistors and considerably smaller than the nominal 100 Ω value or values obtained by post-firing [5].
2) The other samples, on the other hand, display to various extents higher apparent sheet resistance values, this effect varying from moderate for DP 6146 (sample B) and DP 6145, to very large (DP 6142D, DP 7484, especially for surface resistors). In this latter group, a significant part of the resistors appear as open circuits, which is ascribed to development of an "open" zone at the resistor-termination interface (Figure 4).

[1] Profile name : "Yannick32", maximum temperature nominally 900°C, actually ~875°C.

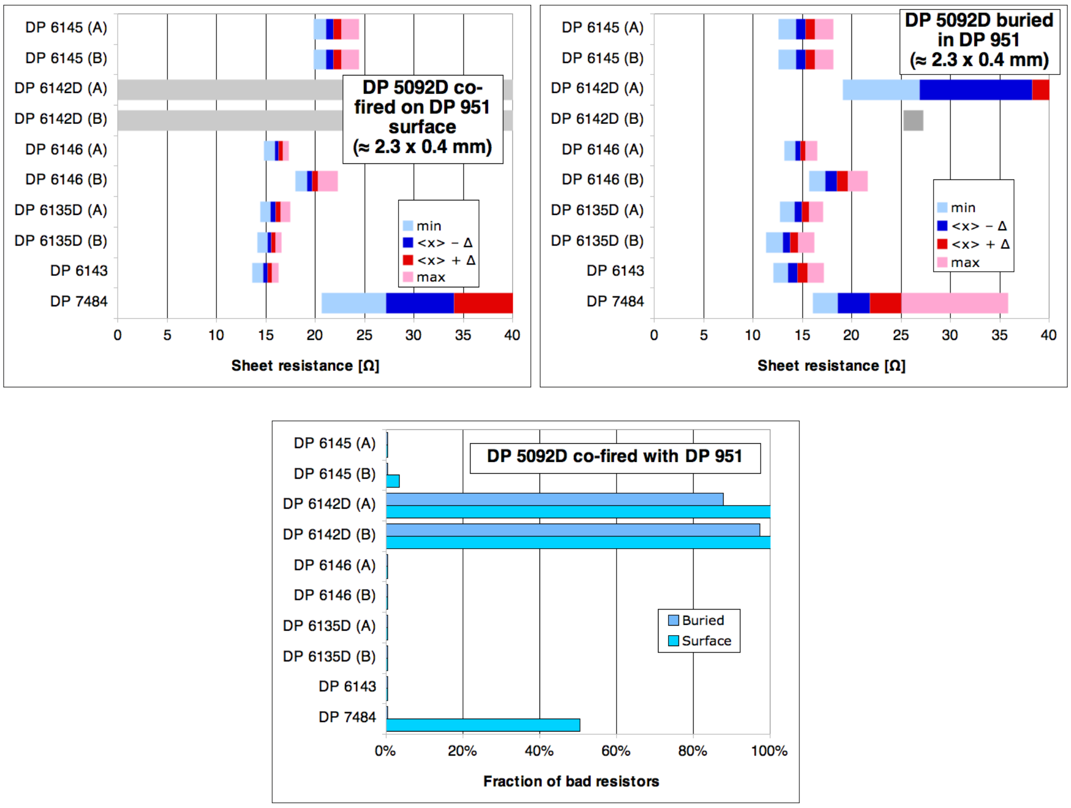

Figure 3. Electrical results, as a function of termination material. Grey bars for the sheet resistance indicate missing or unreliable data due to materials compatibility problems.

Termination material		**Terminations**	**Deformation**
Ag	DP 6142D	Visibly damaged zone (Figure 4)	Slight
	DP 6145	Visibly affected zone; resistor looks "thinner", but still present	Slight
Ag-Pd	DP 6135D	Apparently OK	Strong
	DP 6143	Apparently OK	Very strong
	DP 6146	Apparently OK	Moderate if termination thin; strong if thicker
	DP 7484	Visibly affected zone; especially if small resistor-termination overlap	Moderate

Table 2. Optical characterisation.

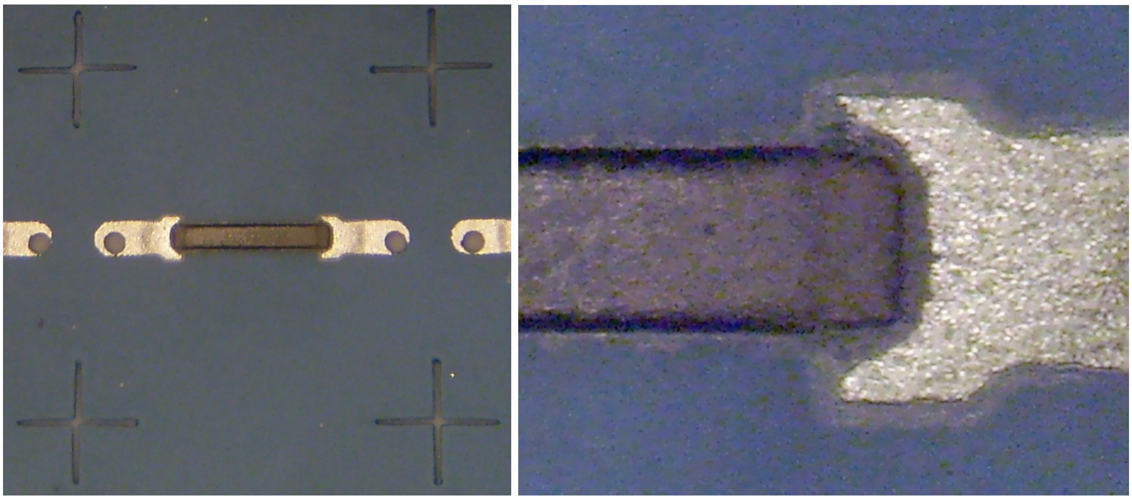

Figure 4. Single surface resistor cofired on DP 951 LTCC (left) and interface between DP 5092D PTC thermistor composition and DP 6145 Ag conductor (right), exhibiting diffusion and disappearance of Ag from the interface.

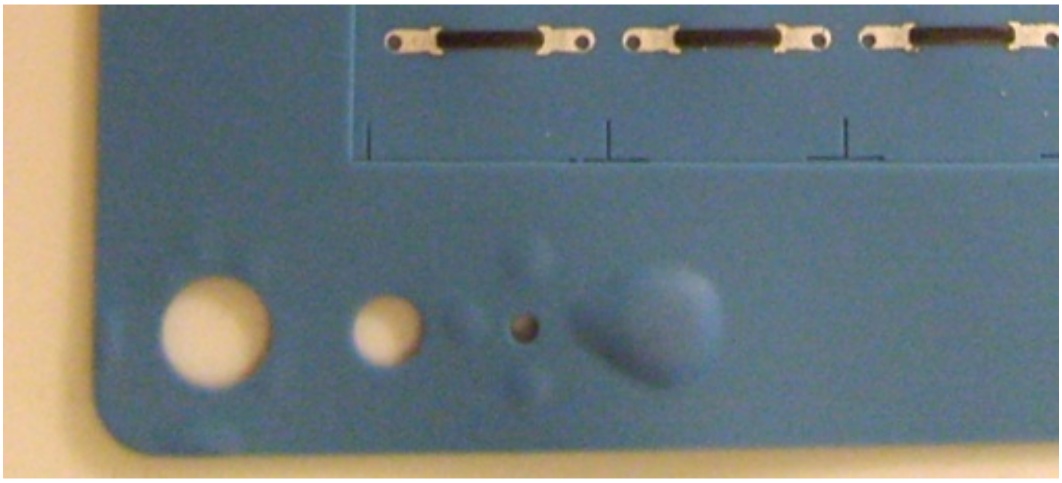

Figure 5. Bubble formation with buried DP 5092D (no contact with metallisation).

Taking a closer look at the (surface) resistors, it appears that, away from the terminations, the DP 5092D material on DP 951 LTCC has a homogeneous and identical aspect for all the samples. Moreover, electrical conductivity of the resistor itself is always apparent even in "defective" resistors, if probed directly on the resistor surface. Therefore, electrical degradation, in the form of increased resistance or of altogether lost electrical contact, is solely due to reactions at the terminations, clearly visible in Figure 4.

The behaviour of buried resistors was in accordance with previous observations: termination problems tend to be less severe, and the swelling (gas bubble formation, Figure 5) accompanying buried DP 5092D material is strongly suppressed by the Ag and Ag-Pd terminations.

Besides electrical integrity, another important criterion for materials selection is the induced deformation (Table 2). It unfortunately appears that the electrically "good" samples, which show no resistance increase due to termination effects and no visible degraded zone there, are those that also exhibit the strongest deformation of the LTCC. This compromise even appears for the same material: among both DP 6146 samples, one (B) due to incidental lower conductor thickness or different effective firing temperature, exhibited lower deformation than the other sample (A), but this also coincided with visibly higher resistance and dispersion, indicative of an onset of degradation at the terminations.

3.2. Thermal characterisation

Five thermistor and one heating resistor were characterised in temperature, by subjecting them to three heating cycles between 22°C and 80°C. The results are given in Table 3. The TCR of the heater is relatively large, but this should not affect the quality of the regulation significantly.
More interestingly, the TCR of the thermistor is quite large (3'435 ppm/K on average vs specified value of 3'000±200 ppm/K [4]), in line with the rather low sheet resistance value (~15 Ω vs 100 Ω nominal), low resistance values tending to give higher TCRs. This gives good sensitivity. Also, reproducibility of the TCR is excellent, ca. 1%.
A more detailed look at linearity yields very small, almost insignificant deviations, less than 0.1%. Reproducibility is also excellent – no apparent drift is observed (Figure 6).

Resistor composition	**DP CF011**	**DP 5092D**
Termination material	DP 6142D (Ag)	DP 6146 (Ag-Pd)
TCR [ppm/K]	+775	+3'435 ± 43
Value C.V.		2.2%
TCR C.V.		1.2%

Table 3. Resistor characteristics, values ± standard deviation. TCR = temperature coefficient of resistance; C.V. = coefficient of variation.

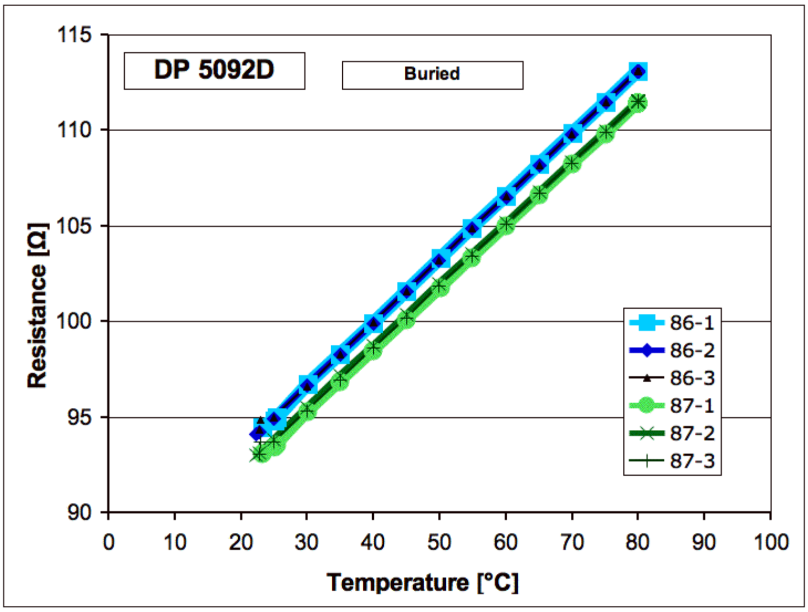

Figure 6. Resistance vs temperature curves of two thermistors.

4. Conclusions

This study allowed to characterise a wide range of Ag & Ag-Pd conductor terminations for co-firing DP 5092D 100 Ω PTC thermistor composition DP 951 LTCC, on the surface or buried inside.

- DP 5092D, cofired with DP 951, exhibits a sheet resistance around 15 Ω, considerably lower than the nominal 100 Ω value, with compatible terminations. This value has remarkably low dispersion, and also depends little on whether the resistor resides on the surface or lies buried within the LTCC.
- Ag-Pd alloy compositions such as DP 6135D, 6143 and 6146 exhibit the best electrical compatibility. These compositions also give rise to the strongest deformations, which should however be only an issue for thin devices.
- However, some degradation is nevertheless possible (DP 6146, sample B). Therefore, the detailed effect of firing conditions (especially peak temperature) and conductor thickness must still be studied in more detail.
- Among Ag compositions, a somewhat compatible one, DP 6145, can be identified, with the advantage of lower deformation. Here also, the abovementioned additional tests are needed to ascertain whether and under which conditions good compatibility and reliability may be obtained.

5. References

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